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## VERIFICATION

I, Kazuo Asahi, of Asahi & Masuda of Nishi-Shinbashi Noa Building 4F & 5F of 18-9, Nishi-Shinbashi 1-chome, Minato-ku, Tokyo 105-0003 Japan do solemnly and sincerely verify that I understand well both Japanese and English languages and that the attached document in English language is full and faithful translation of the copy of Japanese Patent Application No. 9-204697 filed on July 30, 1997.



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COMPOSITION FOR AN ORGANIC EL  
ELEMENT AND METHOD OF  
MANUFACTURING THE ORGANIC EL  
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Specification

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COMPOSITION FOR AN ORGANIC EL ELEMENT AND METHOD OF  
MANUFACTURING THE ORGANIC EL ELEMENT

[CLAIMS]

[Claim 1] A composition for an organic EL element used for forming a pattern by an ink-jet method, which includes as its main component a precursor of a conjugated organic polymer compound for forming a luminescent layer and at least one kind of fluorescent dye for changing the luminescence characteristic of said luminescent layer, wherein the composition for an organic EL element is characterized in that the contact angle of said composition with respect to a material constituting a nozzle surface of a nozzle provided in a head for said ink-jet device for discharging said composition lies in the range of 30 to 170 degrees.

[Claim 2] A composition for an organic EL element used for forming a pattern by an ink-jet method, which includes as its main component a precursor of a conjugated organic polymer compound for forming a luminescent layer and at least one kind of fluorescent dye for changing the luminescence characteristic of said luminescent layer, wherein the composition for an organic EL element is characterized in that the viscosity of the composition is 1cp to 20cp.

[Claim 3] A composition for an organic EL element used for forming a pattern by an ink-jet method, which includes as its main component a precursor of a conjugated organic polymer compound for forming a luminescent layer and at least one kind of fluorescent dye for changing the luminescence characteristic of

said luminescent layer, wherein the composition for an organic EL element is characterized in that the surface tension of said composition is 20dyne to 70dyne.

[Claim 4] A composition for an organic EL element used for forming a pattern by an ink-jet method, which includes as its main component a precursor of a conjugated organic polymer compound for forming a luminescent layer and at least one kind of fluorescent dye for changing the luminescence characteristic of said luminescent layer, wherein the composition for an organic EL element is characterized in that the contact angle of said composition with respect to a material constituting a nozzle surface of a nozzle provided in a head for said ink-jet device for discharging said composition lies in the range of 30 to 170 degrees and the viscosity of the composition is 1cp to 20cp.

[Claim 5] A composition for an organic EL element used for forming a pattern by an ink-jet method, which includes as its main component a precursor of a conjugated organic polymer compound for forming a luminescent layer and at least one kind of fluorescent dye for changing the luminescence characteristic of said luminescent layer, wherein the composition for an organic EL element is characterized in that the contact angle of said composition with respect to a material constituting a nozzle surface of a nozzle provided in a head for said ink-jet device for discharging said composition lies in the range of 30 to 170 degrees and the surface tension of said composition is 20dyne to 70dyne.

[Claim 6] A composition for an organic EL element used for forming a pattern by an ink-jet method, which includes as its main component a precursor of a conjugated organic polymer compound for forming a luminescent layer and at least one kind of

fluorescent dye for changing the luminescence characteristic of said luminescent layer, wherein the composition for an organic EL element is characterized in that the viscosity of the composition is 1cp to 20cp and the surface tension of said composition is 20dyne to 70dyne.

[Claim 7] A composition for an organic EL element used for forming a pattern by an ink-jet method, which includes as its main component a precursor of a conjugated organic polymer compound for forming a luminescent layer and at least one kind of fluorescent dye for changing the luminescence characteristic of said luminescent layer, wherein the composition for an organic EL element is characterized in that the contact angle of said composition with respect to a material constituting a nozzle surface of a nozzle provided in a head for said ink-jet device for discharging said composition lies in the range of 30 to 170 degrees, the viscosity of the composition is 1cp to 20cp, and the surface tension of said composition is 20dyne to 70dyne.

[Claim 8] The composition for an organic EL element as set forth in any one of Claims 1-7, wherein said luminescence characteristic is a maximum wavelength of light absorption.

[Claim 9] The composition for an organic EL element as set forth in any one of Claims 1-8, wherein said precursor includes a precursor of a polyparaphenylene vinylene or its derivative.

[Claim 10] The composition for an organic EL element as set forth in any one of Claims 1-9, wherein said fluorescent dye includes rhodamine or rhodamine derivative.

[Claim 11] The composition for an organic EL element as set forth in any one of Claims 1-9, wherein said fluorescent dye includes distyrylbiphenyl and its derivative.

[Claim 12] The composition for an organic EL element as set forth in any one of Claims 1-9, wherein said fluorescent dye includes coumarin or coumarin derivative.

[Claim 13] The composition for an organic EL element as set forth in any one of Claims 1-9, wherein said fluorescent dye includes tetraphenylbutadiene (TPB) or tetraphenylbutadiene derivative.

[Claim 14] The composition for an organic EL element as set forth in any one of Claims 1-9, wherein said fluorescent dye includes quinacridone or quinacridone derivative.

[Claim 15] The composition for an organic EL element as set forth in any one of Claims 1-14, wherein said precursor and said fluorescent dye exist in the state of being dissolved or dispersed into a polar solvent.

[Claim 16] The composition for an organic EL element as set forth in any one of Claims 1-15, wherein the amount of said fluorescent dye to be added is 0.5 to 10wt% with respect to a solid component of the precursor of said conjugated organic polymer compound.

[Claim 17] The composition for an organic EL element as set forth in any one of Claims 1-16, wherein said composition includes a lubricant.

[Claim 18] A method of manufacturing an organic EL element by using the composition for an organic EL element in any one of claims 1-17, which is characterized by including the steps of forming a pattern by jetting said composition from a head by an ink-jet method; and forming a luminescent layer by polymerizing said precursor in said composition by heat treatment.

[Detailed Description of the Invention]

[0001]

[Technical Field to which the Invention Belongs]

The present invention relates to a composition for an electroluminescent (EL) element, and more particularly to a composition for an organic EL element.

[0002]

[Prior Art]

An organic EL element has a constitution in which a cathode and an anode are connected to the opposite sides of a solid thin film containing a fluorescent organic compound. When a voltage is applied across the electrodes, electrons and holes are injected into the thin film, and they migrate according to the generated electric field and recombine with each other. At the time, excitons are produced by the energy liberated upon the recombination, and energy (in the form of fluorescence or phosphorescence) is emitted when the excitons are returned to the ground state thereof. This phenomenon is called electroluminescence.

[0003]

The characteristic of an organic EL element resides in the point that a high luminance surface emission of 100 to 100,000cd/m<sup>2</sup> is possible at a low voltage of not more than 10V. Further, the use of organic compounds affords the excellent advantage of an unlimited possibility as to the selection of raw materials, which cannot be found in other material systems. In other words, by appropriate selection of the kinds of the fluorescent materials, it is possible to realize emission of any visible rays ranging from blue color to red color.

[0004]

Here, a luminescent layer bears an important part for the

luminescence efficiency and the stability of the element. In the past, doping of high efficiency fluorescent dyes into the luminescent layer has been carried out in order to improve the luminescence efficiency and change the emission wavelength.

#### [0005]

The conventional organic EL element mainly uses a low molecular dye (host material), and it is formed into a thin film to form a laminated type organic thin film EL element. Such an organic thin film EL material is formed by adding a fluorescent dye to a low molecular weight host material, for example, an element obtained by adding a fluorescent dye such as perylene or distyrylbiphenyl or the like to a host material such as aluminum quinolynol complex ( $\text{Alq}_3$ ) or distyrylbiphenyl or the like is mentioned.

#### [0006]

In order to form such a low molecular dye into a thin film, a vapor deposition method has been so far employed. However, it is difficult to obtain a homogeneous defect-free thin film by the vapor deposition method, and the formed thin film has a problem in its stability and strength. Specifically, there is a problem in that when increasing the temperature of the element, crystallization and cohesion of organic molecules occur and the crystallized spot can not make contact with the electrode, causing a nonluminous spot (so-called dark spot). Further, it can not be said that the vapor deposition method is an effective method for manufacturing an element, because it takes long time to form several organic layers using the vapor deposition method.

#### [0007]

##### [Problem to be Solved by the Invention]

It is an object of the present invention to provide a

composition for an organic EL element and a method of manufacturing such an organic EL element which can perform a high precision patterning in a simple manner and in a short time, achieve an optimization of a film design and a luminescence characteristic easily, and facilitate an adjustment of a luminescence efficiency, as well as form a thin film having excellent durability.

[0008]

[Means for Solving the problem]

Such an object is achieved by the present invention as follows from (1) to (18).

[0009]

(1) A composition for an organic EL element used for forming a pattern by an ink-jet method, which includes as its main component a precursor of a conjugated organic polymer compound for forming a luminescent layer and at least one kind of fluorescent dye for changing the luminescence characteristic of the luminescent layer, wherein the composition for an organic EL element is characterized in that the contact angle of the composition with respect to a material constituting a nozzle surface of a nozzle provided in a head for the ink-jet device for discharging the composition lies in the range of 30 to 170 degrees.

[0010]

(2) A composition for an organic EL element used for forming a pattern by an ink-jet method, which includes as its main component a precursor of a conjugated organic polymer compound for forming a luminescent layer and at least one kind of fluorescent dye for changing the luminescence characteristic of the luminescent layer, wherein the composition for an organic EL

element is characterized in that the viscosity of the composition is 1cp to 20cp.

[0011]

(3) A composition for an organic EL element used for forming a pattern by an ink-jet method, which includes as its main component a precursor of a conjugated organic polymer compound for forming a luminescent layer and at least one kind of fluorescent dye for changing the luminescence characteristic of the luminescent layer, wherein the composition for an organic EL element is characterized in that the surface tension of the composition is 20dyne to 70dyne.

[0012]

(4) A composition for an organic EL element used for forming a pattern by an ink-jet method, which includes as its main component a precursor of a conjugated organic polymer compound for forming a luminescent layer and at least one kind of fluorescent dye for changing the luminescence characteristic of the luminescent layer, wherein the composition for an organic EL element is characterized in that the contact angle of the composition with respect to a material constituting a nozzle surface of a nozzle provided in a head for the ink-jet device for discharging the composition lies in the range of 30 to 170 degrees and the viscosity of the composition is 1cp to 20cp.

[0013]

(5) A composition for an organic EL element used for forming a pattern by an ink-jet method, which includes as its main component a precursor of a conjugated organic polymer compound for forming a luminescent layer and at least one kind of fluorescent dye for changing the luminescence characteristic of the luminescent layer, wherein the composition for an organic EL

element is characterized in that the contact angle of the composition with respect to a material constituting a nozzle surface of a nozzle provided in a head for the ink-jet device for discharging the composition lies in the range of 30 to 170 degrees and the surface tension of the composition is 20dyne to 70dyne.

[0014]

(6) A composition for an organic EL element used for forming a pattern by an ink-jet method, which includes as its main component a precursor of a conjugated organic polymer compound for forming a luminescent layer and at least one kind of fluorescent dye for changing the luminescence characteristic of the luminescent layer, wherein the composition for an organic EL element is characterized in that the viscosity of the composition is 1cp to 20cp and the surface tension of the composition is 20dyne to 70dyne.

[0015]

(7) A composition for an organic EL element used for forming a pattern by an ink-jet method, which includes as its main component a precursor of a conjugated organic polymer compound for forming a luminescent layer and at least one kind of fluorescent dye for changing the luminescence characteristic of the luminescent layer, wherein the composition for an organic EL element is characterized in that the contact angle of the composition with respect to a material constituting a nozzle surface of a nozzle provided in a head for the ink-jet device for discharging the composition lies in the range of 30 to 170 degrees, the viscosity of the composition is 1cp to 20cp, and the surface tension of the composition is 20dyne to 70dyne.

[0016]

(8) The composition for an organic EL element as set forth in any one of (1)-(7) in the above, wherein the luminescence characteristic is a maximum wavelength of light absorption.

[0017]

(9) The composition for an organic EL element as set forth in any one of (1)-(8) in the above, wherein the precursor includes a precursor of a polyparaphenylene vinylene or its derivative.

[0018]

(10) The composition for an organic EL element as set forth in any one of (1)-(9) in the above, wherein the fluorescent dye includes rhodamine or rhodamine derivative.

[0019]

(11) The composition for an organic EL element as set forth in any one of (1)-(9) in the above, wherein the fluorescent dye includes distyrylbiphenyl and its derivative.

[0020]

(12) The composition for an organic EL element as set forth in any one of (1)-(9) in the above, wherein the fluorescent dye includes coumarin or coumarin derivative

[0021]

(13) The composition for an organic EL element as set forth in any one of (1)-(9) in the above, wherein said fluorescent dye includes tetraphenylbutadiene (TPB) or tetraphenylbutadiene derivative.

[0022]

(14) The composition for an organic EL element as set forth in any one of (1)-(9) in the above, wherein the fluorescent dye includes quinacridone or quinacridone derivative.

[0023]

(15) The composition for an organic EL element as set forth in any one of (1)-(14) in the above, wherein the precursor and the fluorescent dye exist in the state of being dissolved or dispersed into a polar solvent.

[0024]

(16) The composition for an organic EL element as set forth in any one of (1)-(15) in the above, wherein the amount of the fluorescent dye to be added is 0.5 to 10wt% with respect to a the component of the precursor of the conjugated organic polymer compound.

[0025]

(17) The composition for an organic EL element as set forth in any one of (1)-(16) in the above, wherein the composition includes a lubricant.

[0026]

(18) A method of manufacturing an organic EL element by using the composition for an organic EL element in any one of (1)-(17) in the above, which is characterized by including the steps of forming a pattern by jetting the composition from a head by an ink-jet method; and forming a luminescent layer by polymerizing the precursor in the composition by heat treatment.

[0027]

[Embodiments of the invention]

Hereinbelow, a composition for an organic EL element according to the present invention will be described in detail.

[0028]

The composition for an organic EL element of the present invention is a composition for an organic EL element which is used for forming a pattern by an ink-jet method, which includes a

precursor of a conjugated organic polymer compound (hereinafter, referred to as "precursor") for mainly forming a luminescent layer and at least one kind of fluorescent dye for changing the emission characteristic of the luminescent layer.

[0029]

The precursor of a conjugated organic polymer compound means a material which can be formed into a thin film as a composition for the organic EL element together with a fluorescent dye and the like and then produced a conjugated organic polymer EL layer by heating and hardening as shown, for example, by the following chemical formula (I). For example, when the precursor is a sulfonium salt, an elimination of the sulfonium group is caused by a heat treatment, thereby being turned into the conjugated organic polymer compound.

[0030]

[Chemical Formula (I)]

[0031]

Such a conjugated organic polymer compound exhibits a strong fluorescence in the solid state, and is capable of forming a homogeneous solid super thin film. Further, the conjugated organic polymer compound has an excellent formability, and has a high adhesion to an ITO electrode. In addition, such a precursor of the compound can form a sturdy conjugated polymer film after hardening, so, since a precursor solution can be adjusted to a desired viscosity suitable for ink-jet pattern formation as described later before heating and hardening, it is possible to form an optimum thin film easily in a short time.

[0032]

As for example of such a precursor, a precursor of PPV (poly(para-phenylene vinylene)) or its derivative is preferable.

Since the precursor of PPV (poly(para-phenylene vinylene)) or its derivative is soluble in water or an organic solvent and such a precursor can be polymerized, it is possible to obtain a thin film having high optical quality. Further, since PPV possess a strong fluorescence and it is a conductive polymer in which  $\pi$ -electron of double bond is delocalized on the polymer chain, it is possible to obtain an organic EL element having high performance.

[0033]

As for example of such a precursor of PPV (poly(para-phenylene vinylene)) or PPV derivative, for example, as shown in the chemical formula (II), PPV (poly(para-phenylene vinylene)) precursor, MO-PPV (poly(2,5-dimethoxy-1,4-phenylene vinylene)) precursor, CN-PPV (poly(2,5-bis(hexyloxy)-1,4-phenylene-(1-cyanovinylene))) precursor, MEH-PPV (poly[2-methoxy-5-(2'-ethylhexyloxy)]-para-phenylene vinylene) precursor or the like is mentioned.

[0034]

[Chemical Formula (II)]

[0035]

The precursor of PPV or the PPV derivative is soluble in water as described above, and is polymerized by heating after formation of a film thereof to form a PPV layer.

[0036]

The content of the precursor represented by the PPV precursor is preferably 0.01 to 10.0wt%, and more preferably 0.1 to 5.0wt%, with respect to the total amount of the composition. If the amount of the precursor to be added is too small, it is insufficient for formation of the conjugated polymer film, and if the amount of the precursor to be added is too much, there is a

case that the viscosity of the composition becomes high, and therefore it is not suitable for pattern formation with high precision by the ink-jet method.

[0037]

Further, the composition for an organic EL element according to the present invention includes at least one kind of fluorescent dye. This makes it possible to change the luminous property of the luminescent layer and is also effective as a means, for example, for improving the luminous efficiency of the luminescent layer or for changing the maximum wavelength of light absorption (emitted color) of the luminescent layers.

[0038]

Namely, a fluorescent dye can be utilized not only as a material for the luminescent layers but also as a material for the coloring matter which exhibits the luminescence function by itself. This is because almost all of the energy of the excitons which are generated by the recombination of carriers on the molecules of a conjugated organic polymer compound can be transferred onto the molecules of the fluorescent dye. In this case, the current quantum efficiency of the EL element is also increased, because the luminescence arises only from the molecules of the fluorescent dye which have a high fluorescence quantum efficiency. Accordingly, by the addition of a fluorescent dye into the composition for the organic EL element, the luminescence spectrum of the luminescent layer becomes the spectrum of the fluorescent dye, thereby enabling to be effective as a means for changing the emitted color.

[0039]

Now, what is meant here by the current quantum efficiency is a scale for considering the luminescence performance according

to the luminescence function, and it is defined by the following expression.

[0040]

$\eta_E$  = energy of emitted photon / inputted electrical energy  
In this way, by changing the maximum wavelength of light absorption by doping of a fluorescent dye, it becomes possible, for example, to emit the three primary colors of red, green and blue, thereby enabling to obtain a full color display device.

[0041]

Further, the doping of the fluorescent dye also makes it possible to improve extremely the luminous efficiency of the EL element.

[0042]

As for a fluorescent dye, rhodamine or a rhodamine derivative for generating red emitted color is preferable. These fluorescent dyes are soluble in aqueous solutions because they are low molecules, and have a high compatibility with PPV, so that a luminescent layer with a good uniformity and good stability can be formed easily.

[0043]

As for examples of such fluorescent dyes, rhodamine B, rhodamine B base, rhodamine 6G, and rhodamine 101 perchlorate and the like can be mentioned, and a mixture of two or more of them can be employed.

[0044]

Further, as for a fluorescent dye, quinacridone and its derivative for generating green emitted color are preferable. Since these fluorescent dyes are low molecules likewise the red fluorescent dyes, they are soluble in aqueous solutions and have a high compatibility with PPV, so that the luminescent layer can

be formed easily.

[0045]

As for the fluorescent dye, distyrylbiphenyl and its derivative for generating blue emitted color are preferable. Since These fluorescent dyes are low molecules likewise the red fluorescent dyes, they are soluble in water and alcohol mixed solutions and have a high compatibility with PPV, so that the luminescent layer can be formed easily.

[0046]

Further, as for other fluorescent dyes for generating blue emitted color, coumarin and its derivative are preferable. These fluorescent dyes are soluble to aqueous solutions because they are low molecules likewise the red fluorescent dyes, and have high compatibility with PPV, so that the luminescent layer can be formed easily.

[0047]

As for examples of such fluorescent dyes, coumarin, coumarin-1, coumarin-6, coumarin-7, coumarin 120, coumarin 138, coumarin 152, coumarin 153, coumarin 311, coumarin 314, coumarin 334, coumarin 337, coumarin 343 and the like can be mentioned.

[0048]

Further, as for other fluorescent dyes for generating blue emitted color, tetraphenylbutadiene (TPB) or TPB derivative are preferable. These fluorescent dyes are soluble in aqueous solutions because they are low molecules in the same manner as the above-mentioned fluorescent dyes for the red luminescent layer, and have high compatibility with PPV, so that the luminescent layer can be formed easily.

[0049]

These fluorescent dyes as described above can be

selectively used alone or in the form of a mixture of two or more of them.

[0050]

The amount of these fluorescent dyes to be added to the solid component of the precursor of the conjugated organic polymer compound are preferably 0.5 to 10wt%, and more preferably 1.0 to 5.0wt%. If the amount of the fluorescent dye to be added is too much, it becomes difficult to maintain the weather resistance and the durability of the luminescent layer. On the other hand, the amount of the fluorescent dye to be added is too little, it is impossible to obtain sufficiently the effect obtained by adding the fluorescent dye as described above.

[0051]

Moreover, it is preferable that the precursor and the fluorescent dye are dissolved or dispersed into a polar solvent. The polar solvent is capable of readily dissolving or homogeneously dispersing the precursor and the fluorescent dye or the like, and this makes it possible to prevent the solid component in the organic EL composition from being stuck at the nozzle hole of ink-jet device and to prevent the nozzle hole from being clogged with the solid component. Further, it also contributes to maintain the high contact angle of the ink at the nozzle hole so that it is possible to prevent the bend in the jetted direction of the ink.

[0052]

As for examples of the polar solvent, water; water compatible alcohol such as methanol, ethanol and the like; organic solvents such as N,N-dimethylformamide (DMF), N-methylpyrrolidone (NMP), dimethylimidazoline (DMI), dimethylsulfoxide (DMSO) and the like; and inorganic solvents can

be mentioned, and a mixture of two or more of these solvents can be employed as required.

[0053]

In addition, it is preferred that a lubricant is added in the composition for the organic EL element. This makes it possible to prevent effectively the composition from being dried and solidified at the ink-jet nozzle hole. As for examples of such a lubricant, polyhydric alcohols such as glycerin and diethylene glycol and the like can be mentioned, and it is also possible to use a mixture of two or more of them.

[0054]

The amount of the lubricant to be added is preferably about 5 to 20wt% with respect to the total amount of the composition.

[0055]

Moreover, other additives and film stabilizing materials may also be added. For example, stabilizer, viscosity adjusting agent, anti-aging agent, pH adjusting agent, antiseptic agent, resin emulsion, leveling agent or the like may be added.

[0056]

The composition for an organic EL element including the above-mentioned precursor and fluorescent dye is used for forming a pattern by an ink-jet method.

[0057]

Here, the manufacturing method of an EL element by the ink-jet printing means a method for forming pixels with at least one color selected from the three primary colors comprising red, green, and blue and their intermediate colors by discharging the aforementioned composition, which is dissolved or dispersed into a solvent, as a discharge liquid from the head.

[0058]

According to the ink-jet printing method, a fine pattern formation can be carried out simply in a short time. In addition, depending on the discharging amount, the adjustment of film thickness can be done easily so that film properties, emission balance and luminance and the like can be controlled readily and arbitrarily.

[0059]

Such a composition has characteristics as described below.

[0060]

It is preferable that the compositions have a contact angle of 30 to 170 degrees, and more preferably, 35 to 65 degrees, with respect to the material constituting the nozzle surface 251 of the nozzle which discharges the composition and is provided in the head of the ink-jet device. When the compositions have the contact angle within this range, it is possible to carry out a precise patterning by suppressing the bend in the jetted direction of the composition.

[0061]

Namely, if the contact angle is less than 30 degrees, wettability of the composition with respect to the material constituting the nozzle surface increases, so that there is a case that the composition adheres asymmetrically to the periphery of the nozzle hole at the time of discharging the composition. In this case, since an attraction acts between the composition adhered to the nozzle hole and the composition to be discharged, the composition is discharged by a nonuniform force, so that a situation so-called "bend in the jetted direction" in which the composition is unable to reach the target position arises, and further, the occurrence frequency of the bend in the jetted direction also tends to increase. On the other hand, if the

contact angle exceeds 170 degrees, the interaction between the composition and the nozzle hole becomes a minimum and the shape of the meniscus at the tip of the nozzle is unstable, which results in the difficulty in the control of the amount and the timings of discharge of the composition.

[0062]

In the above descriptions, what is meant here by the bend in the jetted direction refers to a situation in which, when the composition is discharged from the nozzle, the point of impact of a dot deviates from the target position by more than  $50 \mu\text{m}$ . Further, the occurrence frequency of the bend in the jetted direction is defined as the time until the bend in the jetted direction occurs after a continuous discharge of the composition is started at the frequency of 7200Hz. The bend in the jetted direction is generated principally in the case where the wettability of the nozzle hole is nonuniform and by the clogging of the nozzle hole due to the adhesion of the solid component of the composition, and such bend in the jetted direction can be eliminated by cleaning the head. In this connection, more frequent head cleaning is required for more frequency of occurrence of the bend in the jetted direction, and this is the composition to deteriorate the manufacturing efficiency of the EL element by the ink-jet method. On the practical level, it is required that an occurrence frequency of the bend in the jetted direction is more than 1000 seconds or more.

[0063]

By preventing such bend in the jetted direction from occurring, it becomes possible to carry out a highly fine patterning, moreover with high precision.

[0064]

Further, it is preferable that the viscosity of the composition is 1cp to 20cp, and more preferably 2cp to 4cp. If the viscosity of the composition is less than 1cp, the contents of the precursor and the fluorescent dye in the material are too small, so that the formed luminescent layer cannot exhibit luminescence power sufficiently. On the other hand, if it exceeds 20cp, it becomes impossible to discharge the composition smoothly from the nozzle, so that, it becomes difficult to carry out patterning unless otherwise the specification of the ink-jet device is changed, for example, by enlarging the diameter of the nozzle hole and so forth. In addition, when the viscosity of the composition is high, precipitation of the solid components in the composition tends to occur, thus leading to an increased occurrence frequency of clogging of the nozzle hole.

[0065]

Furthermore, it is preferable that the composition has the surface tension of 20 dyne to 70 dyne, and more preferably 25 dyne to 40 dyne. By setting the surface tension to such a range, it is possible to suppress the bend in the jetted direction and thereby hold the occurrence frequency of the bend in the jetted direction to a low level, in the same manner as the case of the above-mentioned contact angle. If the surface tension is less than 20 dyne, the wettability of the composition with respect to the material constituting the nozzle surface increases, so that the bend in the jetted direction occurs and occurrence frequency tends to increase in the same manner as the case of the above-mentioned contact angle. On the other hand, if it exceeds 70 dyne, the shape of the meniscus at the tip of the nozzle is not stable, which results in the difficulty in the control of the amount and the timings of discharge of the composition.

[0066]

Further, although the composition of an organic EL element of this invention is a composition which satisfies the numerical range for at least one of the characteristics including the contact angle, the viscosity and the surface tension described above, a composition which satisfies the requirements for arbitrary combination of two characteristics, and further a composition which satisfies requirements for all these characteristics. This makes the composition be more suitable one for the ink-jet method.

[0067]

The method of manufacturing the organic EL element according to the present invention is characterized by including a step of forming a pattern by jetting out the above-mentioned composition for an EL element from the nozzle hole of the head for the ink-jet method, and a step of forming a luminescent layer by polymerizing the above-mentioned precursor of the above-mentioned composition by a heat treatment. According to this method, since the precursor of the conjugated organic polymer compound, which is prior to polymerizing, is used as a material, the degrees of freedom in the selection for the contact angle, viscosity and surface tension of the composition material become large. Accordingly, since it is possible to discharge an arbitrary amount of the composition solution to an arbitrary position, a precise pattern formation can be performed and the emission characteristics and the film properties of the luminescent layer can be easily controlled.

[0068]

The structure of the head of the ink-jet device used in the manufacturing method of the organic EL element according to the

present invention is shown in Fig. 2 and Fig. 3.

[0069]

The head 10 for the ink-jet device is provided with, for example, a stainless steel nozzle plate 11 and a vibration plate 13, and they are coupled via partitioning members (reservoir plates) 15.

[0070]

Between the nozzle plate 11 and the vibration plate 13, a plurality of spaces 19 and a liquid storage 21 are formed by means of the reservoir plates 15. The inside of the respective spaces 19 and the liquid storage 21 are filled with the composition according to the present invention, and the spaces 19 are in communication with the liquid storage 21 through supply ports 23.

[0071]

Further, a nozzle hole 25 is provided in the nozzle plate 11 for discharging the composition in a jet-form from the spaces 19. On the other hand, a hole 27 is formed in the vibration plate 13 for supplying the composition to the liquid storage 21.

[0072]

Further, on a surface of the vibration plate 13 which is opposite to the surface that faces with the spaces 19, piezoelectric elements 29 are attached at positions corresponding to the positions of the respective spaces 19.

[0073]

The piezoelectric element 29 is located between a pair of electrodes 31, and when the piezoelectric element 29 is energized, the piezoelectric element 29 is bent to outside projectingly, and at the same time, the vibration plate 13 connected with the piezoelectric element 29 is bent to outside in a body with

element 29. This increases the volume of the space 19. Therefore the composition whose volume is equivalent to the increased volume in the space 19 is flowed in from the liquid storage 21 through the supply ports 23.

[0074]

Next, when the piezoelectric element 29 is stopped energizing, both the piezoelectric element 29 and the vibration plate 13 are brought back to the initial form. Consequently, since the volume of the space 19 is returned to the former volume, the pressure of the composition in the space 19 becomes high and the composition is jetted from the nozzle hole 25 toward the substrate.

[0075]

Now, a water repellent layer 26 is provided at the peripheral part of the nozzle hole 25 in order to prevent the bend in the jetted direction of the composition and to prevent clogging of the hole by the composition.

[0076]

Namely, as shown in Fig.3, a water repellent layer 26 is provided, for example, which is constructed from a layer to which eutectic tetrafluoroethylene-nickel is plated.

[0077]

Using the head described above, it is possible to form organic luminescent layers respectively by discharging the compositions corresponding to, for example, the three primary colors red, blue, and green according to predetermined patterns, thereby enabling to form the pixels.

[0078]

According to the ink-jet method as described above, the arbitrary amount of the composition to be discharged, the number

of times of jetting the composition and the forms of patterns can be adjusted easily and handily, so that the luminescent characteristics of the luminescent layers and film characteristics, for example, film thickness and the like can be controlled.

[0079]

Further, in the thin film formed in a manner described above, there is no problem such as the dark spot which appears in the vapor deposition method, so that it is possible to obtain an excellent EL element.

[0080]

[Embodiment]

In what follows, the present invention are described in details with reference to the actual examples.

[0081]

1. Forming into the pattern of Organic EL Element

(Example 1)

The composition for an organic EL element made from the composition shown in Table 1 was discharged from the nozzle for the ink-jet device by the method as shown in Fig. 1 to form patterns.

[0082]

[Table 1]

[0083]

As shown in Fig. 1, on the glass substrate 104, ITO transparent pixel electrodes 101, 102 and 103 were formed by means of photolithography so as to form respectively a pattern having a pitch of  $100\mu\text{m}$  and a thickness of  $0.1\mu\text{m}$ .

[0084]

Then, a black resist polymer 105 which buries the spaces

between the ITO transparent pixel electrodes and acts not only as a layer for cutting off the light but also as a wall for preventing ink drop are formed by photolithography. Each of the black resist was designed so as to have a width of  $20\mu\text{m}$  and a thickness of  $2.0\mu\text{m}$ , respectively.

[0085]

Further, the above-mentioned compositions were discharged from the head 110 of the ink-jet device 109.

[0086]

(Example 2)

A composition was discharged from the head 110 of the ink-jet device 109 in the same way as in Example 1 except that distyrylbiphenyl was added as a blue fluorescent dye instead of the rhodamine B.

[0087]

[Table 2]

[0088]

(Example 3)

A composition was discharged from the head 110 of the ink-jet device 109 in the same way as in Example 1 except that quinacridone was added as a green fluorescent dye instead of the rhodamine B.

[0089]

[Table 3]

[0090]

(Example 4)

A composition was discharged from the head 110 of the ink-jet device 109 in the same way as in Example 1 except that coumarin 6 was added as a blue fluorescent dye instead of the rhodamine B and glycerin of 3 wt% and diethylene glycol of 12 wt%

were added as a lubricant.

[0091]

[Table 4]

[0092]

(Example 5)

A composition was discharged from the head 110 of the ink-jet device 109 in the same way as in Example 1 except that TPB (tetraphenylbutadiene) was added as a blue fluorescent dye instead of the rhodamine B.

[0093]

[Table 5]

[0094]

(Example 6)

A composition was discharged from the head 110 of the ink-jet device 109 in the same way as in Example 1 except that CN-PPV precursor which emits red color was used instead of PPV precursor without adding any red fluorescent dye and that quinacridone of 0.0075 wt% was added as a green fluorescent dye.

[0095]

[Table 6]

[0096]

(Comparative Example 1)

A composition was discharged from the head 110 of the ink-jet device 109 in the same way as in Example 1 except that a composition which includes aqueous solution of PPV precursor of 50wt%; glycerin of 20wt% and diethylene glycol of 20 wt% for lubricants; and DMF of 10 wt% for polar solvent; and quinacridone as a green fluorescent dye instead of the rhodamine B was used, as shown in Table 7.

[0097]

[Table 7]

[0098]

(Comparative Example 2)

A composition was discharged from the head 110 of the ink-jet device 109 in the same way as in Example 1 except that a composition which includes DMF of 75wt% for a polar solvent and no lubricant and coumarin 6 as a blue fluorescent dye instead of rhodamine B was used, as shown in Table 8.

[0099]

[Table 8]

[0100]

(Comparative Example 3)

A composition was discharged from the head 110 of the ink-jet device 109 in the same way as in Example 1 except that a composition which includes aqueous solution of PPV precursor of 50wt%; glycerin of 10wt% and diethylene glycol of 10wt% for lubricants; and DMF of 30wt% for polar solvent was used as shown in Table 9.

[0101]

[Table 9]

[0102]

2. Evaluation of Physical Properties and Discharge Characteristic of the Composition

The contact angle, viscosity and surface tension of the compositions of Examples 1 to 6 and Comparative Examples 1 to 4 were measured.

[0103]

In addition, these compositions were discharged from the nozzle hole of an ink-jet printer (made by EPSON Co., Ltd. Model No. MJ-500C, the material constituting the nozzle surface is a

water repellent layer to which eutectic tetrafluoroethylene-nickel is plated) and were formed into patterns.

[0104]

The occurrence frequency of the bend in the jetted direction of the composition and the occurrence frequency of clogging of the nozzle hole at the time of discharging the composition were investigated.

[0105]

① Contact Angle

The contact angle for each composition with respect to the material constituting the nozzle surface was measured in accordance with the measurement method of the contact angle as stipulated in JIS K3211.

[0106]

② Viscosity

The viscosity of each composition at 20°C was measured using Type E viscometer.

[0107]

③ Surface Tension

For the surface tension "γ" of each composition, the measurement was carried out by means of the drip method as follows.

[0108]

When a liquid is dripped gently from the mouth of a circular tube with flat cut plane, a droplet falls as its weight overcomes the surface tension. If the mass of the droplet is called "m", the force pulling it downward is "mg", and the force pulling it upward is  $2\pi r\gamma$  ("r" is the outer radius of the mouth of the pipe). Based on the relationship between the two forces, the surface tension "γ" of the composition was determined by

measuring "m".

[0109]

④ Occurrence Frequency of Bend in Jetted Direction

When the composition was continuously discharged (at a frequency of 7200Hz), a time required until the bend in the jetted direction occurred was measured.

[0110]

⑤ Occurrence Frequency of Clogging of Nozzle Hole

When the composition was continuously discharged (at a frequency of 7200Hz), a time required until the nozzle hole was leaded to be impossible to discharge the composition because of the clogging of the nozzle hole by the precipitated solid component of the composition, was measured.

[0111]

The results of these experiments are shown in Table 10.

[0112]

[Table 10]

[0113]

As shown in Table 10, each of the compositions in Examples 1 to 6 has lower values of the occurrence frequency of the bend in the jetted direction and the occurrence frequency of the clogging of the nozzle hole, and the occurrence frequencies indicated by the values were within the range of the practical level sufficiently.

[0114]

In particular, when all of the characteristics including the contact angle, the viscosity and the surface tension of the compositions fall within the ranges specified by this invention, the occurrence frequency of the bend in the jetted direction and the occurrence frequency of the clogging of the nozzle hole were

low level, so that it was found that they were extremely excellent compositions.

[0115]

In contrast, when the composition in Comparative Example 1 was discharged, the occurrence frequency of the bend in the jetted direction and the occurrence frequency of the clogging of the nozzle hole were high level since the composition of Comparative Example 1 has a tendency that the solid component in the composition is likely to stick at the periphery of the nozzle hole, so that it was found this composition can not be practically used. The composition in Comparative Example 2 has a large value for the occurrence frequency of the bend in the jetted direction, so that it was found that it is not suitable for fine pattern formation. Further, for the composition in Comparative Example 3, the clogging of the nozzle occurred in a short time as soon as the discharge of the composition was started, so that it could hardly discharge the composition.

[0116]

3. Formation of Organic EL Element

Next, the layers which were formed of the respective compositions in Examples 1 to 6 were undergone heat treatment under a nitrogen atmosphere at 150 °C for 4 hours and were polymerized the precursors in the compositions, thereby forming luminescent layers 106 (red), 107 (green) and 108 (blue) which emit red, green and blue respectively.

[0117]

Then, by carrying out a vacuum deposition of aluminum quinolynol complex to which no doping was carried out, the electron transfer layer 111 having a thickness of 0.1  $\mu$  m was formed. This electron transfer layer 111 facilitates injection

of the electrons from the cathode, and contributes to the prevention of electrode quenching by keeping the luminescent portions away from the cathode to establish a better contact with the cathode.

[0118]

Finally, an Al - Li reflection electrode 112 having a thickness of  $0.8 \mu\text{m}$  was formed as the counter electrode by the deposition method, and thereby manufacturing the organic EL element.

[0119]

#### 4. Evaluation of the Luminescence Characteristics and the Film Characteristics of the Luminescent Layers

The luminescence characteristics and the film characteristics of the respective luminescent layers which were formed of the respective composition in Examples 1 to 6 described above, were evaluated according to the following methods.

[0120]

##### ⑥ Luminescence Starting Voltage

A prescribed voltage was applied to the element, and the applied voltage at which a luminance of  $1 \text{ cd/m}^2$  is observed was defined as the luminescence starting voltage [V<sub>th</sub>].

[0121]

##### ⑦ Luminescence Life

The initial luminance after a stabilization treatment has been carried out was set to 100%, and the changes in the luminance of the EL element were measured under the condition that the elements were kept in continuous luminescence by supplying a constant current of standard waveform, wherein the luminescence life is defined as the time until the luminance drops to 50% of the initial luminance.

[0122]

Now, the drive conditions for this experiment were set at 40 °C for room temperature, 23% for humidity and 20 mA/cm<sup>2</sup> for current density.

[0123]

⑧ **Luminance (Brightness)**

The luminance was measured at which the current density was set to 20 mA/cm<sup>2</sup>.

[0124]

⑨ **Wavelength at Maximum Absorption**

The wavelength at maximum absorption for each luminescent layer was measured.

[0125]

⑩ **Stability in Film Formation**

After heating the luminescent layer at 200 °C for 60 minutes, conditions for occurrence of cracks and deformation and the like in the respective luminescent layers were observed by a microscope.

[0126]

The results of the evaluation are shown in Table 11.

[0127]

[Table 11]

[0128]

As can be seen from Table 11, the luminescent characteristics and the film properties of all of the luminescent layers formed using the compositions of Examples 1 to 6 were excellent. Namely, it was possible to obtain organic EL elements free from dark spots and have high luminance and long life.

[0129]

In the above, the compositions for an organic EL element

and the methods of manufacturing the organic EL elements have been described for respective embodiments illustrated. However, the present invention is not limited to these embodiments, and it may further include a step of inserting arbitrary functional intermediate layers between the respective layers. Moreover, the fluorescent dye to be added for changing the luminescent characteristics is not limited to those of the embodiments as long as they can be dissolved and dispersed homogeneously into a polar solvent.

[0130]

Furthermore, the material constituting the nozzle surface of the ink-jet printer is not limited to those of the embodiments described above.

[0131]

**[Effect of the Invention]**

As described in the above, according to the compositions for an organic EL element of the present invention, a rational design of the organic EL element is possible through wide ranging selection of luminescent materials. Namely, by assorting a conjugated organic polymer compound and a fluorescent dye, various kinds of light for display can be obtained, so that it is possible to realize a full color display. Accordingly, it is possible to develop designs for various organic EL elements with high luminance and long life.

[0132]

According to the above-mentioned compositions of the present invention, it is possible to employ the ink-jet method to the formation of a luminescent layer. In that case, it is possible to carry out easily a high precise pattern formation in a short time without the occurrence of the bend in the jetted

direction of the composition and the occurrence of the clogging of the nozzle hole and the like. In addition, the optimizations of the film design and the luminescent characteristics can be readily achieved, thereby enabling to form a luminescent layer which is capable of easy adjustment of luminescence efficiency and have excellent durability.

[0133]

Further, since the precursor which is polymerized by heating and hardening is included as the luminescent layer material, conditions such as contact angle, viscosity and surface tension can be freely set, and then the conditions suitable for a discharge liquid used for the ink-jet method can be easily adjusted.

[0134]

Further, according to the method of manufacturing the composition for the organic EL element, conditions such as the film thickness, the number of dots and the like can be adjusted arbitrarily, so that the luminescence characteristics of the luminescent layers can be controlled easily and the size and the pattern of the luminescent element can also be set arbitrarily.

[Brief Description of the Drawings ]

[Fig. 1]

This figure is a sectional view which shows steps performed in the method of manufacturing the organic EL element of the present invention.

[Fig. 2]

This figure is a perspective view which shows an example of a configuration of a printer head of an ink-jet device which is used for manufacturing the organic EL element.

[Fig. 3]

This figure is a sectional view of the nozzle part of the head for the ink-jet printer device shown Fig. 2.

[Description of Symbols]

- 10 Head for the ink-jet device
- 11 Nozzle plate
- 13 Vibration plate
- 15 Reservoir plate
- 19 Space
- 21 Liquid storage
- 23 Supply port
- 25 Nozzle hole
- 26 Water repellent layer
- 27 Hole
- 29 Piezoelectric element
- 31 Electrode
- 101 Transparent pixel electrode
- 102 Transparent pixel electrode
- 103 Transparent pixel electrode
- 104 Glass substrate
- 105 Polymer black resist
- 106 Organic luminescent layer (red)
- 107 Organic luminescent layer (green)
- 108 Organic luminescent layer (blue)
- 109 Ink-jet printer device
- 110 Head
- 111 Electron transfer layer
- 112 Counter electrode
- 251 Nozzle surface

[Document Name] Abstract

[Summary]

[Problem] In forming a pattern by an ink-jet method, a high precision pattern formation can be carried out simply in a short time without the bend in the jetted direction of the composition or the clogging of the nozzle hole.

[Solving Means] The composition for an organic EL element used for forming a pattern by an ink-jet method, which comprises as its main component a precursor of a conjugated organic polymer compound for forming luminescent layers and at least one kind of fluorescent dye for changing the luminescence characteristic of the luminescent layer, satisfies at least one of the conditions including a contact angle of 30 to 170 degrees with respect to a material constituting a nozzle surface of a head for an ink-jet device used for the ink-jet method, a viscosity of 1 to 20cp and a surface tension of 20 to 70dyne. As for the precursor in the composition, for example, polyphenylene vinylene or its derivative can be mentioned. As for the fluorescent dye, for example, rhodamine B, distyrylbiphenyl, coumarin, tetraphenylbutadiene, quinacridone, and their derivatives can be mentioned.

[Selected Drawings] Fig. 1

(Example 1)

Composition	Name of Material	Amount of Content (wt%)
Precursor	aqueous solution of PPV precursor (1.5wt%) (mixed solution of water/methanol=5/95)	25
Wetting Agent	glycerin	5
	diethylene glycol	10
Polar Solvent	DMF (N,N-dimethyl formamide)	60
Fluorescent Dye	rhodamine B	0.0075 (Ratio of Solid Portion of Precursor 2wt%)

Table 2

(Example 2)

Composition	Name of Material	Amount of Content (wt%)
Precursor	aqueous solution of PPV precursor (1.5wt%) (mixed solution of water/methanol=5/95)	2 5
Wetting Agent	glycerin	5
Polar Solvent	diethylene glycol	1 0
Fluorescent Dye	DMF (N,N-dimethyl formamide)	6 0
	distyrylbiphenyl	0. 0075 (Ratio of Solid Portion of Precursor 2 wt%)

Table 3  
(Example 3)

Composition	Name of Material	Amount of Content (wt%)
Precursor	aqueous solution of PPV precursor (1.5wt%) (mixed solution of water/methanol=5/95)	25
Wetting Agent	glycerin	5
Polar Solvent	DMF (N,N-dimethyl formamide)	60
Fluorescent Dye	quinacridone	0.0075 (Ratio of Solid Portion of Precursor 2wt%)

(Example 4)

Composition	Name of Material	Amount of Content (wt%)
Precursor	aqueous solution of PPV precursor (1.5wt%) (mixed solution of water/methanol=5/95)	25
Wetting Agent	glycerin diethylene glycol	3 12
Polar Solvent	DMF (N,N-dimethyl formamide)	60
Fluorescent Dye	coumarin 6	0.0075 (Ratio of Solid Portion of Precursor 2 wt%)

(Example 5)

Table 5

Composition	Name of Material	Amount of Content (wt%)
Precursor	aqueous solution of PPV precursor (1.5wt%) (mixed solution of water/methanol=5/95)	2 5
Wetting Agent	glycerin	5
	diethylene glycol	1 0
Polar Solvent	DMF (N,N-dimethyl formamide)	6 0
Fluorescent Dye	TBP (tetraphenylbutadiene)	0. 0075 (Ratio of Solid Portion of Precursor 2wt%)

Table 6  
(Example 6)

Composition	Name of Material	Amount of Content (wt%)
Precursor	aqueous solution of CN-PPV precursor (1.5wt%) (mixed solution of water/methanol=5/95)	25
Wetting Agent	glycerin diethylene glycol	5 10
Polar Solvent	DMF (N,N-dimethyl formamide)	60
Fluorescent Dye	quinacridone	0.0075 (Ratio of Solid Portion of Precursor 2wt%)

Table 7

(Comp. Ex. 1)

Composition	Name of Material	Amount of Content (wt%)
Precursor	aqueous solution of PPV precursor (1.5wt%) (mixed solution of water/methanol=5/95)	5 0
Wetting Agent	glycerin	2 0
	diethylene glycol	2 0
Polar Solvent	DMF (N,N-dimethyl formamide)	1 0
Fluorescent Dye	quinacridone	0. 0075 (Ratio of Solid Portion of Precursor 1 wt%)

(Comp. Ex. 2)

Table 8

Composition	Name of Material	Amount of Content (wt%)
Precursor	aqueous solution of PPV precursor (1.5wt%) (mixed solution of water/methanol=5/95)	25
Wetting Agent	glycerin	0
Polar Solvent	diethylene glycol	0
Fluorescent Dye	DMF (N,N-dimethyl formamide) coumarin 6	75 0.0075 (Ratio of Solid Portion of Precursor 2wt%)

(Comp. Ex. 3)

Table 9

Composition	Name of Material	Amount of Content (wt%)
Precursor	aqueous solution of PPV precursor (1.5wt%) (mixed solution of water/methanol=5/95)	5 0
Wetting Agent	glycerin	1 0
	diethylene glycol	1 0
Polar Solvent	DMF (N,N-dimethyl formamide)	3 0
Fluorescent Dye	rhodamine B	0. 0075 (Ratio of Solid Portion of Precursor 1wt%)

(Result of Experiment)

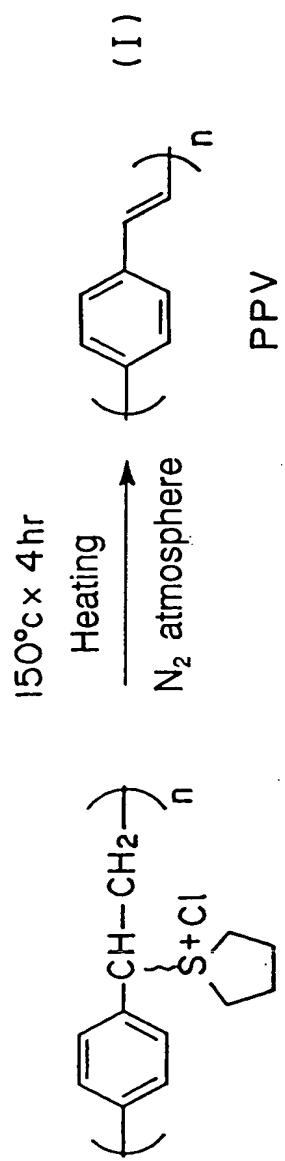
Table 10

Composition	Viscosity [cp]	Surface Tension [dyne/cm]	Contact Angle [°]	Occurrence Frequency of Bend in Jetted Direction (sec)	Occurrence Frequency of Clogging (sec)
Example 8	3. 72	32. 9	59	3. 000	more than 10. 000
Example 9	3. 60	30. 1	60	2. 800	more than 10. 000
Example 10	3. 62	39. 6	40	2. 800	more than 10. 000
Example 11	2. 80	31. 6	38	2. 900	more than 10. 000
Example 12	3. 58	33. 2	36	3. 000	9. 000
Example 13	3. 86	33. 6	64	2. 800	9. 000
Comp. Ex. 3	30. 3	18. 5	20	100	1. 000
Comp. Ex. 4	0. 9	90	175	200	more than 10. 000
Comp. Ex. 5	25	15	28	Impossible to discharge composition	

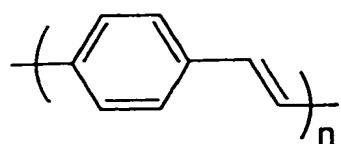
Table 11

	Luminescence Starting Voltage [V <sub>th</sub> ]	Luminescence Life [hr]	Luminance [cd/m <sup>2</sup> ]	Wavelength at Maximum Absorption [nm]	Stability in Film Formation
Example 8	3. 0	5000	2000	620	○
Example 9	4. 5	4900	1800	420	○
Example 10	4. 2	4800	1700	530	○
Example 11	4. 3	4900	1900	480	○
Example 12	4. 0	4800	1900	430	○
Example 13	5. 6	4500	1850	530	○

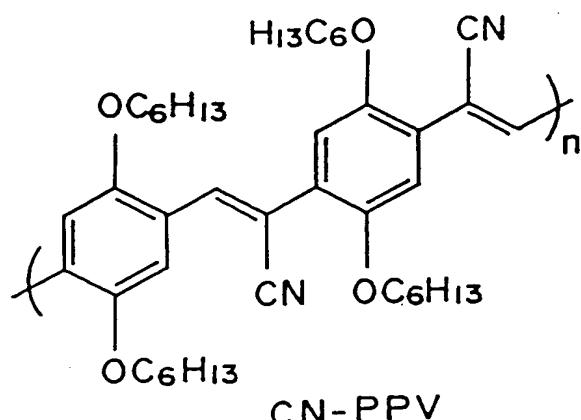
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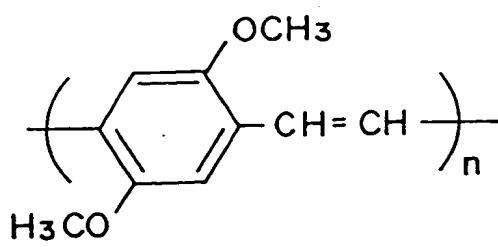
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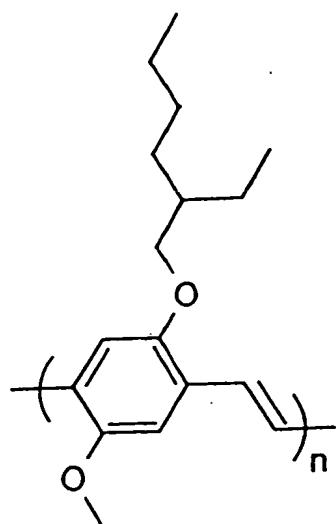
PPV



CN-PPV



## MO-PPV



## MEH-PPV

(II)